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IS 12685 (1989): Pesticides - Pendimethalin, Technical [FAD 1: Pesticides and Pesticides Residue Analysis]

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IS 12685 : 1989
(Reaffirmed 2004)

Indian Standard

PESTICIDES — PENDIMETHALIN, TECHNICAL — SPECIFICATION

भारतीय मानक

कीटनाशक – पेन्डीमेथालीन, तकनीकी – विशिष्टि

UDC 632'951 PEN

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NEW DELHI 110002

November 1989

Price Group **2**

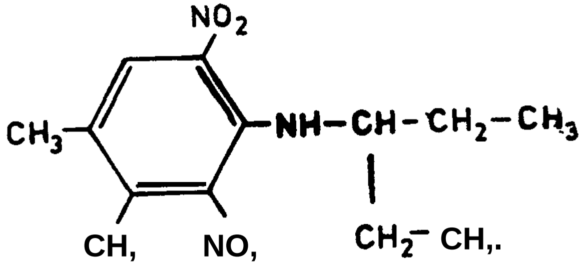
Pest Control Sectional Committee, AFCDC 6

FOREWORD

This Indian Standard was adopted by the Bureau of Indian Standards on 24 May 1989, after the draft finalized by the Pest Control Sectional Committee had been approved by the Agricultural and Food Products Division Council.

Pendimethalin is a herbicide.

Pendimethalin is the accepted common name by the International Organization for Standardization (ISO) for the herbicidal chemical *N*-(1-ethylpropyl) -2, 6-dinitro-3, 4-xylidine or *N*-(1-ethylpropyl) -3, 4-dimethyl-2, 6-dinitrobenzenamine. The empirical and structural formulae and the molecular mass of pendimethalin are as indicated below:

Empirical Formula	*Structural Formula*	*Molecular Mass*
$C_{13}H_{19}N_3O_4$		*281’3*

In the preparation of this standard, due consideration has been given to the provisions of the ***Insecticides Act, 1968*** and the Rules framed thereunder. However, this standard is subject to the restrictions imposed under these, wherever applicable.

For the purpose of deciding whether a particular requirement of this standard is complied with, the final value, observed or calculated, expressing the result of a test or analysis, shall be rounded off in accordance with IS *2* : 1960 ‘Rules for rounding off numerical values (***revised***)’. The number of significant places retained in the rounded off value should be the same as that of the specified value in this standard.

Indian Standard

PESTICIDES — PENDIMETHALIN, TECHNICAL — SPECIFICATION

1 SCOPE

1.1 This standard prescribes the requirements and the methods of sampling and test for pendimethalin, technical.

2 REFERENCES

IS No.	*Title*
IS 6940 : 1982	Methods of test for pesticides and their formulations (*first revision*)
IS 8190 (Part 1) : 1988	Requirements for packing of pesticides: Part 1 Solid pesticides (*second revision*)
IS 10946 : 1984	Methods of sampling for technical grade pesticides

3 REQUIREMENTS

3.1 Description

Pendimethalin, technical, shall be odourless, in the form of yellow to brown crystalline solid, free from any extraneous impurities.

3.2 The material shall also comply with the requirements specified in Table 1.

4 PACKING AND MARKING

4.1 Packing

The material shall be packed in mild steel containers with low density polyethylene liner of thickness not less than 0'062 mm, in addition, complying with general packing requirements as given in 2 of IS 8190 (Part 1) : 1988.

4.2 Marking

The container shall bear legibly and indelibly the following information and any other information as is necessary under the *Insecticides Act, 1968* and Rules.

a) Name of the material;

b) Name of the manufacturer;

c) Batch number;

d) Date of manufacture;

e) Pendimethalin content, percent (*m/m*);

f) Net mass of contents; and

g) Minimum cautionary notice worded as in the *Insecticides Act, 1968* and Rules.

Table 1 Requirements for Pendimethalin, Technical

(*Clauses 3.2 and 6.1*)

Sl No.	Characteristic	Requirement	Method of Test, Ref to: Annex of This Standard	Method of Test, Ref to: *Cl No.* of IS 6940 : 1982
(1)	(2)	(3)	(4)	(5)
i)	Pendimethalin content, percent by mass, *Min*	90·0	A	—
ii)	Material insoluble in acetone, percent by mass, *Max*	1·0	—	9
iii)	Acidity (as H_2SO_4), percent by mass, Max	0·5	—	11·3
	Or			
	Alkalinity (as NaOH), percent by mass, *Max*	0·5	—	11.3
iv)	Melting point	54 to 58°C	—	6

NOTE — Two consecutive determinations of melting point in the same sample shall not differ by more than 1'0°C.

5 SAMPLING

5.1 Representative samples of the material shall be drawn as prescribed in IS 10946 : 1984.

6 TESTS

6.1 Tests shall be carried out in accordance with **3.1, 3.2** and by the methods referred to in col 4 and 5 of Table 1.

6.2 Quality of Reagents

Unless specified otherwise, pure chemicals and distilled water shall be employed in tests.

NOTE — 'Pure chemicals' shall mean chemicals, that do not contain impurities which affect the result of analysis.

ANNEX A

[*Table* 1, *item* (i)]

DETERMINATION OF PENDIMETHALIN CONTENT

A-l GENERAL

A-l.1 Either of the two methods, namely, gas chromatographic (GLC) method or spectrophotometric method may be used for determination of pendimethalin content. However, GLC method shall be the referee method.

A-2 GAS CHROMATOGRAPHIC METHOD

A-2.1 Principle

The pendimethalin content is determined using an internal standard, on a gas chromatograph equipped with a flame ionization detector.

A-2.2 **Apparatus**

A-2.2.1 *Gas Liquid Chromatograph*

It shall be equipped with an or column injection system, flame ionization detector and shall be coupled to a strip chart recorder. An electronic integrator may also be used.

a) *Detector*: Flame ionization detector (FID)

b) *Column*: Borosilicate glass; 1‘3 m long; 6 mm OD; 3 mm ID, packed with 3 percent OV-225 on gas chrom Q, 100-120 mesh

c) *Glass wool*: Silane treated

d) *Temperature*

1) Column : 220°C
2) Injection : 230°C
3) Detector : 270°C

e) *Gas* — *Flow Rate*

1) Nitrogen (Carrier) : 30 ml/minute
2) Hydrogen Fuel : 30 ml/minute
3) Air : 300 ml/minute

NOTE — Operating parameters as well as attenuation and chart speed shall be adjusted to obtain optimum response and reproducibility.

A-2.3 Reagents

A-2.3.1 *Standard Reference Pendimethalin,* of known purity or secondary standard of 90 percent plus purity.

A-2.3.2 *Internal Standard,* p-terphenyl (p-diphenyl benzene).

A-2.3.3 *Acetone,* glass distilled, analytical reagent grade.

A-2.3.4 *Chloroform,* glass distilled, analytical reagent grade.

A-2.4 **Procedure**

A-2.4.1 *Preparation of Internal Standard Solution*

Prepare 1 percent (*m/v)* solution of p-terphenyl p-diphenyl benzene) in chloroform. This solution will remain stable for four weeks if kept tightly sealed under refrigeration. Allow the solution to attain room temperature before use.

A-2.4.2 *Preparation of Reference Standard Solution*

Weigh accurately, 110 mg of pendimethalin reference standard into a 50-ml volumetric flask. Add by pipette, exactly 5 ml of the internal standard solution to the flask. Add chloroform to the mark. This solution will remain stable for four weeks if the flask is kept sealed and under refrigeration. Allow the solution to warm to room temperature before use.

A-2.4.3 *Preparation of Sample Solution*

A-2.4.3.1 *For technical material and liquid formulation*

Weigh accurately sample equivalent to 110 mg of pendimethalin into a 50-ml volumetric flask. Add by pipette, 5 ml internal standard solution. Dilute to the mark with chloroform and mix well.

A-2.4.3.2 *For granular formulation*

Weigh accurately a quantity of granules equivalent to approximately 500 mg pendimethalin in a 50-ml flask. Add 25 ml chloroform and shake continuously for 15 minutes. Filter through sintered glass (G 3 or G 4) funnel. Repeat the extraction in the same manner twice with 25 ml chloroform and transfer the extracts in a 100-ml volumetric flask. Make up the volume to 100-ml. Take 20 ml of this solution in a volumetric flask, add 5 ml of internal standard solution, make up the volume with chloroform and mix well.

A-2.4.4 *Analysis of Sample*

Check the chromatograph for thermal and flow equilibrium. Inject alternatively 2 μl of standard solution (A-2.4.2) and 2 μl of test solution (A-2.4.3). Measure the peak areas of standard, sample and internal standard solutions injected. Determine the Rf value for each injection.

A-2.4.5 *Calculation*

$$\text{Pendimethalin content, percent by mass} = \frac{m_1 \times R_2}{m_2 \times R_1} \times P \times D$$

where

m_1 = mass, in g, of pendimethalin standard;

m_2 = mass, in g, of sample taken for the test;

$R_1 = \dfrac{\text{peak area of pendimethalin standard}}{\text{peak area of internal standard}}$

$R_2 = \dfrac{\text{peak area of sample}}{\text{peak area of internal standard}}$

P = percent purity of pendimethalin standard; and

D = dilution factor (5 for granules).

A-3 SPECTROPHOTOMETRIC METHOD

A-3.1 Principle

Pendimethalin is separated from known visible absorbing contaminants by thin-layer chromatography (TLC). The band corresponding to the pendimethalin is scraped from the TLC plate, extracted from the silica gel, and quantitatively estimated spectrophotometrically in the visible range (approximately at 425 nm).

A-3.2 Apparatus

A-3.2.1 *Spectrophotometer*

A-3.2.2 *Thin-Layer Chromatography (TLC) Plates, 200* mm × 200 mm, coated with silica gel G or equivalent.

A-3.2.3 *Lambda pipette 500, µl*

A-3.2.4 Centrifuge tubes, stoppered

A-3.2.5 Developing tank

A-3.3 **Reagents**

A-3.3.1 *Silica Gel G or Equivalent*

A-3.3.2 Chloroform, analytical reagent grade.

A-3.3.3 *Petroleum Ether,* 30 — 60°C boiling point.

A-3.3.4 *Methyl Alcohol,* analytical reagent grade.

A-3.3.5 *Triethylamine,* analytical reagent grade.

A-3-3.6 *Pendimethalin Reference Standard,* of *known* purity or secondary standard of 90 percent (m/m), minimum purity.

A-3.4 Procedure

A-3.4.1 *Preparation of Sample Solution*

A-3.4.1.1 *For technical and EC*

Weigh a quantity of sample containing an equivalent of 300±20 mg of pendimethalin in an accurately weighed, dry, 25-ml volumetric flask with stopper, Care shall be taken to avoid contact of the sample with the ground glass joint of the flask. Replace the stopper and note the exact weight. Add chloroform to the mark, replace the stopper and mix well by shaking.

A-3.4.1.2 *For granular formulation*

Weigh granular formulation equivalent to 600± 20 mg of pendimethalin in a volumetric flask and extract successively three times each with 25-ml of chloroform by shaking for about 15 minutes. Filter each extract through sintered glass filter (G3/G4). Transfer the combined filtrate in a 50-ml volumetric flask and add chloroform to the mark.

A-3.4.2 *Method*

A-3.4.2.1 Streak 500 µl with the help of a lambda pipette across a TLC plate, positioning the streak about 3 cm from the bottom edge. Care shall be taken to keep the band width within 2 cm and all the liquid shall be removed out of the pippette by moving it slowly back and forth across the plate, Allow the plate to dry in air.

A-3.4.2.2 Place the plate in a tank previously equilibrated with petroleum ether; triethylamine (95 : 5 *v/v*) added to a depth of about 1 cm. Remove the plate when the solvent has developed to within 3 cm from top. Allow the plate to dry in air. Return the plate to the same tank and develop a second time.

A-3.4.2.3 The plate is now placed with one side edge on a piece of butter paper and slanted over the paper with the silica coating downward. Using a small spatula with a straight, sharp end, scrape the lower half of the yellow pendimethalm band (with downward strokes) so that the silica falls on the paper. The remaining fine powder may be removed with a rubber policeman. The other half of the band is removed by turning the plate around and repeating the scraping procedure. Remove an equivalent portion of the blank plate by the same technique. Carefully transfer the silica gel into a glass stoppered 100-ml volumetric flask.

A-3.4.2.4 Add 50 ml of methanol with the help of a pipette to the powder in the flask, stopper and mix intermittently for about 14 to 15 minutes. Centrifuge a 10 to 12 ml portion of the solution in stoppered centrifuge tube and take out with a pipette, accurately 5·0 ml of the clear supernatant liquid into a 25-ml volumetric flask. Make up to mark with methanol and mix well. Determine the absorbance *(A)* of the sample at the wavelength of maximum absorption for pendimethalin at 425 nm.

A.3.4.2.5 Weigh accurately 300 mg of pendimethalin reference standard into a 25-ml volumetric flask, dilute to the mark with methanol and mix well. Follow the procedure given in A-3.4.2.1 to A-3.4.2.4. Determine the absorbance (*B*) of the standard solution at 425 nm.

A-3.4.3 *Calculation*

$$\text{Pendimethalin content, percent by mass} = \frac{A}{B} \times \frac{m}{M} \times D \times P$$

where

A = absorbance of sample solution at 425 nm;

m = mass, in g, of pendimethalin reference standard taken for the preparation of standard solution;

D = factor, 25 for *EC* and 50 for *Granules*;

P = percent purity of the pendimethalin reference standard:

B = absorbance of standard solution at 425 nm; and

A4 = mass, in g, of the material taken for the test.

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